Synthesis and X-Ray Structures of Some Mono- and Binuclear Ruthenium Complexes with Bisphosphine Ligands

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Summary. The dinuclear complexes $\{RuCp^*(\mu\text{-}Cl)\}_2(\mu\text{-}dppm)$ (**1**) and $\{RuCp^*(\mu\text{-}Cl)\}_2$ $(\mu\text{-}dppe)$ (**3**) are obtained by reacting $[RuCp^*(\mu^3\text{-}Cl)]_4$ with *dppm*, and *dppe*, respectively. **1** is readily oxidized with $AgCF_3SO_3$, instead of chloride abstraction, to afford the dinuclear complex $[\{RuCp^*(\mu\text{-}Cl)\}_2(\mu\text{-}dppm)](SO_3CF_3)_2$ (**2**) with two metal centers connected by a single Ru–Ru bond. Under the same conditions, **3** decomposes to several intractable materials. Similarly to **1**, $RuCp^*(dmpe)Cl$ reacts with $AgCF_3SO_3$ to afford the Ru(III) complex $[RuCp^*(dmpe)Cl](SO_3CF_3)$ (**4**) without no halide abstraction. The crystal structures of **2**, **3**, and **4** are presented.

Keywords. Ruthenium; Binuclear complexes; X-Ray analysis; Oxidation; Bisphosphines.

Synthese und Röntgenstrukturanalyse einiger ein- und zweikerniger Rutheniumkomplexe mit Bisphosphinliganden

Zusammenfassung. Die Komplexe $\{RuCp^*(\mu\text{-}Cl)\}_2(\mu\text{-}dppm)$ (**1**) und $\{RuCp^*(\mu\text{-}Cl\}_2(\mu\text{-}dppe)$ (**3**) wurden durch Umsetzung von $[RuCp^*(\mu^3\text{-}Cl)]_4$ mit *dppm* bzw. *dppe* dargestellt. **1** wird durch $AgCF_3SO_3$ zum zweikernigen Komplex $[\{RuCp^*(\mu\text{-}Cl)\}_2(\mu\text{-}dppm)](SO_3CF_3)_2$ (**2**) oxidiert, welcher eine Ru–Ru-Metallbindung aufweist. Unter den gleiche Reaktionsbedingungen zersetzt sich **3** zu undefinierten Produkten. Analog zu **1** reagiert $RuCp^*(dmpe)Cl$ mit $AgCF_3SO_3$ zum Ru(III)-Komplex $[Ru(Cp^*)(dmpe)Cl](SO_3CF_3)$ (**4**) wobei es zu keiner Chloridabspaltung kommt. Von **2**, **3**, und **4** wurden die Kristallstrukturen bestimmt.

Introduction

Since transition metal complexes having free coordination sites feature highly reactive intermediates in many stoichiometric and catalytic transformations of organic molecules [1], their specific generation and detailed study is an attractive topic of basic research. A common approach to such species is removing halide from metal halide complexes by means of Ag^+, Tl^+, or-occasionally-Na^+ salts of weakly basic counter anions such as $CF_3SO_3^-$, PF_6^-, or BPh_4^- in poorly coordinating solvents. We have previously shown that $RuCp^*(dppe)]Cl$ (*dppe* $= Ph_2PCH_2$-

CH_2PPh_2) has a labile chloride ligand which can easily be abstracted by Ag^+ to yield the coordinatively unsaturated species $[RuCp^*(dppe)]^+$ [2, 3]. This compound reacts readily with small molecules such as O_2, H_2, and Cl_2 to afford the complexes $[RuCp^*(dppe)(\eta^2\text{-}O_2]^+$, $[RuCp^*(dppe)(H)_2]^+$, and $[RuCp^*(dppe)\,(Cl)_2]^+$.

In the present work, we describe the syntheses of the mononuclear complex $RuCp^*(dmpe)Cl(dmpe = Me_2PCH_2CH_2PMe_2)$ and the dinuclear complexes $\{RuCp^*(\mu\text{-}Cl)\}_2(\mu\text{-}dppm)$ $(dppm = Ph_2PCH_2PPh_2)$ and $\{RuCp^*(\mu\text{-}Cl)\}_2(\mu\text{-}dppe)$ with either dppm or dppe acting as a bridging ligand. The latter complex appears to be the first ruthenium complex containing dppe as a bridging ligand. The behavior of the individual complexes towards $AgCF_3SO_3$ in the weakly coordinating solvent CH_2Cl_2 has been studied with the objective of generating reactive coordinatively unsaturated species. In addition, crystal structures of some reaction products are presented.

Results and Discussion

Treatment of $[RuCp^*(\mu^3\text{-}Cl)]_4$ with 2 equivalents of dppm in CH_2Cl_2 at room temperature afforded the dinuclear complex $\{RuCp^*(\mu\text{-}Cl)\}_2(\mu\text{-}dppm)$ (1) as an orange-brown solid in 77% isolated yield (Scheme 1). The bisphosphine has to be added slowly in order to avoid the formation of the mononuclear complex $Ru(Cp^*)(dppm)Cl$. Formerly, 1 has been prepared in low yield from the dinuclear complex $\{RuCp^*\}_2(\mu\text{-}Cl_3)$. The NMR spectroscopic properties of 1 are in accordance with reported values [4].
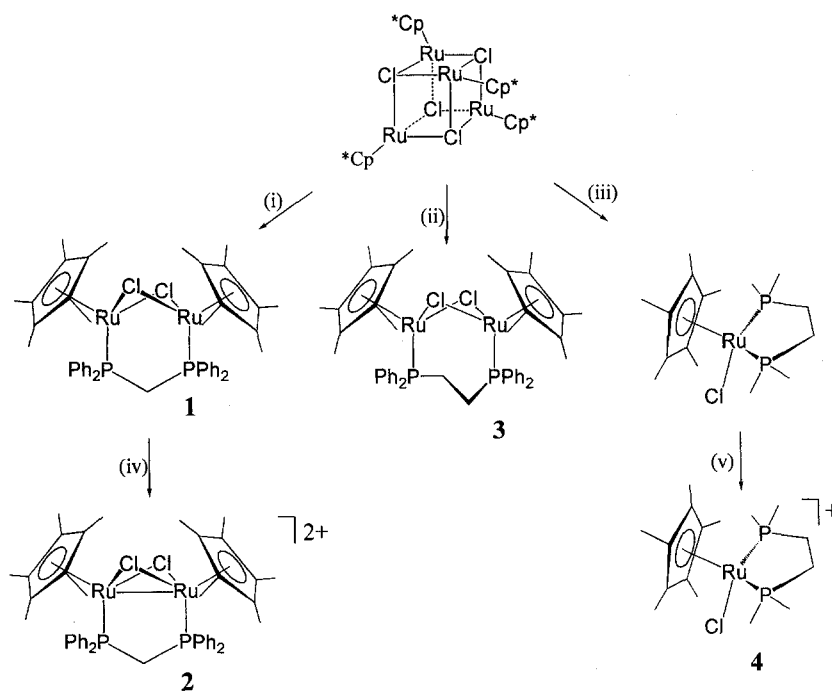

Scheme 1. (i) 2 equiv. of dppm in CH_2Cl_2, room temperature; (ii) 2 equiv. of dppe in CH_2Cl_2, room temperature; (iii) 4 equiv. of dmpe in CH_2Cl_2, room temperature; (iv) 2 equiv. of $AgCF_3SO_3$ in CH_2Cl_2, room temperature; (v) 1 equiv. of $AgCF_3SO_3$ in CH_2Cl_2, room temperature

In an attempt to obtain a coordinatively unsaturated complex, **1** was treated with 2 equivalents of $AgCF_3SO_3$ in CH_2Cl_2 for 1 h. However, instead of acting as a chloride scavenger, the silver cation cleanly oxidized **1** to the dicationic complex $[\{RuCp^*(\mu\text{-Cl})\}_2(\mu\text{-}dppm)](SO_3CF_3)_2$ (**2**) in high yield (Scheme 1). Complex **2** was characterized by a combination of elemental analysis, ^1H, and ^{13}C NMR spectroscopy. In the ^{13}C NMR spectrum, the chemical shifts of the ring carbon atoms appear at 112.5 ppm. This marked low-field shift is indicative of a higher oxidation state of Ru. For comparison, the corresponding carbon resonance in the Ru(II) complex **1** appears at 79.5 ppm. The oxidation of **1** is reversible; upon addition of either Zn or $HBEt_3^-$ to a solution of **2** in CH_2Cl_2, **1** is regained.

Crystal structure of 2

The structure of **2**, as determined by X-ray crystallography, is depicted in Fig. 1. Selected bond distances and bond angles are given in Table 1. The two $RuCp^*$ moieties are joined at a Ru–Ru separation of 2.921(1) Å and are additionally linked by two bridging chlorides and a bridging *dppm* ligand. The Ru–Ru distance is longer than in most dinuclear ruthenium complexes, yet within the range of a Ru–Ru single bond. For comparison, the Ru–Ru distance in the Ru(III) dimer $Ru_2Cl_6(dmpm)_4(dmpm = Me_2PCH_2PMe_2)$ is 2.933 Å [5], whereas in the Ru(I) dimers $[Ru_2(CO)_4Cl(PPh_2)(dppm)]$ [6] and $Ru_2(\mu\text{-l})_2(CO)_4(dppm)$ [7] the Ru–Ru distances are significantly shorter (2.730(1) and 2.707(1) Å, respectively). The Ru–Cl distance is in the range of 2.360(2) to 2.377(2) Å similar to the Ru–Cl distance of the bridging chloride ligand in $[RuCp^*(\mu\text{-Cl})Cl]_2$ (2.366(1) Å [8]. The angles between Cl(1)-Ru(1)-Cl(2) and Cl(1)-Ru(2)-Cl(2) are 103.19(6) and 102.86(6)°, respectively.
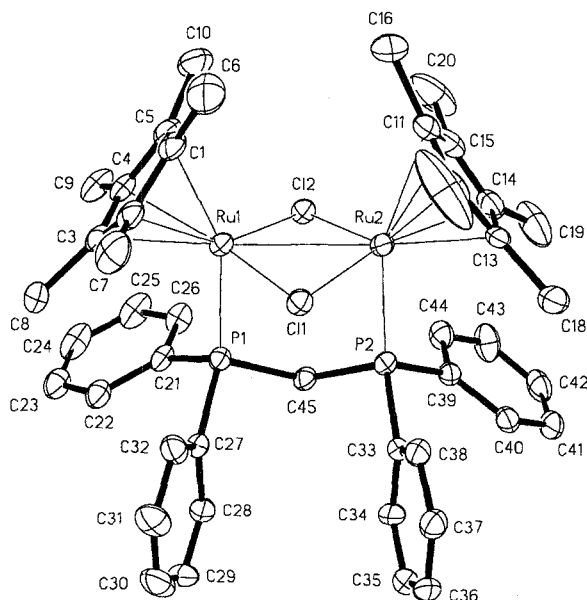
Fig. 1. Structural view of $[\{RuCp^*-(\mu\text{-Cl})\}_2(\mu\text{-}dppm)](SO_3CF_3)_2$ (**2**) showing 30% probability thermal ellipsoids ($CF_3SO_3^-$ omitted for clarity)

Table 1. Selected bond lengths (Å) and angles (°) for $[\{RuCp^*(\mu\text{-Cl})\}_2(\mu\text{-}dppm)](SO_3CF_3)_2$ (2), $\{RuCp^*(\mu\text{-Cl})\}_2(\mu\text{-}dppe)$ (3), and $[Ru(Cp^*)(dmpe)Cl](SO_3CF_3)$ (4)

	2	3	4
Bond lengths			
Ru(1)–Ru(2)	2.921(1)	3.856(1)	
Ru(1)–Cl(1)	2.360(2)	2.497(1)	2.353(1)
Ru(1)–Cl(2)	2.372(2)	2.521(1)	
Ru(1)–P(1)	2.352(2)	2.329(2)	2.322(1)
Ru(1)–C₅(Cp*)	2.241(7)	2.170(5)	2.233(4)
Ru(2)–Cl(1)	2.365(2)	2.506(1)	
Ru(2)–Cl(2)	2.377(2)	2.511(1)	
Ru(2)–P(2)	2.359(2)	2.305(2)	
Ru(2)–C₅(Cp*)	2.236(8)	2.173(5)	
Bond angles			
Cl(1)–Ru(1)–Cl(2)	103.2(1)	79.5(1)	
Cl(1)–Ru(2)–Cl(2)	102.9(1)	79.5(1)	

Dinuclear $R_2P(CH_2)_nPR_2$ bridged ruthenium complexes are not restricted to ligands with $n = 1$, e.g. dppm, but can also be obtained with ligands with $n = 2$. Thus, the reaction of $[RuCp^*(\mu^3\text{-Cl})]_4$ with 2 equivalents of dppe yields the new dinuclear complex $\{RuCp^*(\mu\text{-Cl})\}_2(\mu\text{-}dppe)$ (3) in 60% yield. Characterization was again performed by elemental analysis, ^{1}H, and ^{13}C NMR spectroscopy. The NMR spectra of 3 are similar to those of 1 and are not further discussed. However, all attempts to prepare the divalent complex $[\{RuCp^*(\mu\text{-Cl})\}_2(\mu\text{-}dppe)](SO_3CF_3)_2$ by treatment of 3 with $AgCF_3SO_3$ failed, leading to complete decomposition of the starting material and to the formation of several intractable materials.
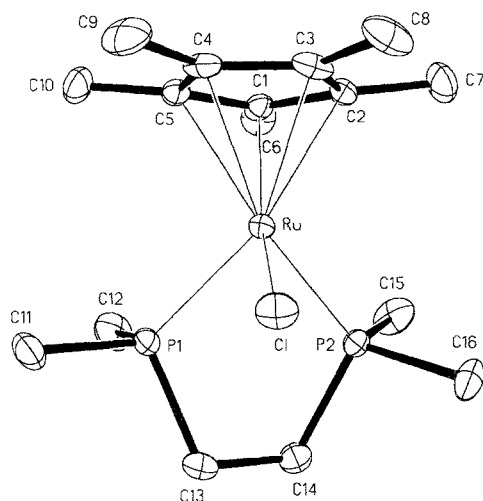
Crystal structure of 3

A structural view of 3 is shown in Fig. 2. Selected bond distances and bond angles are given in Table 1. According to our knowledge, this seems to be the first crystal structure of a dinuclear dppe-bridged ruthenium complex, whereas trinuclear ruthenium complexes featuring a bridging dppe ligand have been reported in the literature [9]. The two RuCp* moieties are linked by two bridging chlorides and a bridging dppm ligand. The Ru–Ru separation is 3.856(1) Å, clearly indicating no Ru–Ru bonding interaction. The Ru–Cl bond distances are in the range of 2.497(1) to 2.521(1) Å, typical for Ru(II) complexes, but are significantly longer than the Ru–Cl distances in 2. The angles between Cl(1)-Ru(1)-Cl(2) and Cl(1)-Ru(2)-Cl(2) are 79.52(4) and 79.54(4)°, respectively.

In contrast to RuCp*(dppe)Cl [2, 3], RuCp*(dmpe)Cl reacts readily with 1 equivalent of $AgCF_3SO_3$ to afford the cationic Ru(III) complex $[Ru(Cp^*)(dmpe)Cl](SO_3CF_3)$ (4) in high yield. Due to the paramagnetic nature of 4, the recording of NMR spectra was precluded and characterization was achieved only by elemental analysis. Presumably, the higher basicity of dmpe relative to dppe accounts for the easy oxidation.
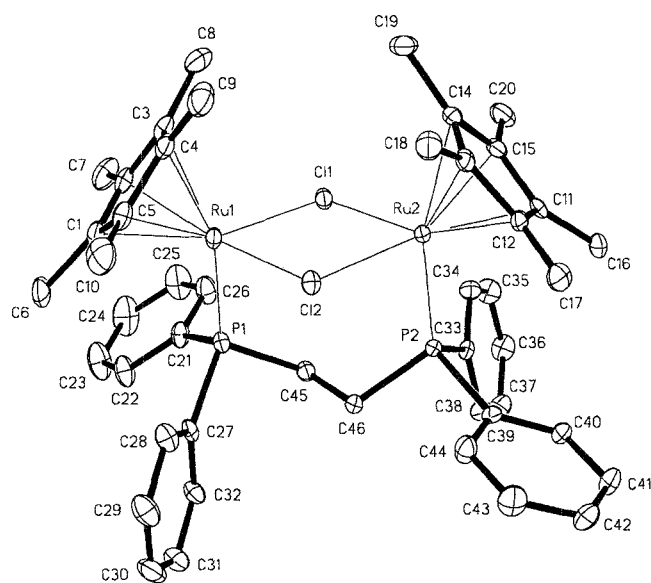
Fig. 2. Structural view of {Ru-Cp*(μ-Cl)}₂(μ-dppe) (**3**) showing 30% probability thermal ellipsoids

Fig. 3. Structural view of [Ru(Cp*)(dmpe)Cl]-(SO₃CF₃) (**4**) showing 30% probability thermal ellipsoids (CF₃SO₃⁻ omitted for clarity)

Crystal structure of **4**

The structure of **4**, as determined by X-ray crystallography, is depicted in Fig. 3. Selected bond distances and angles are given in Table 1. **4** adopts the usual distorted "three-legged" piano stool structure. The Ru–P(1), Ru–P(2), and Ru–Cl distances are 2.322(1), 2.311(1), and 2.353(1) Å, respectively, with a P–Ru–P angle of 79.4(1)°. The mean Ru–C₅(Cp*) distance is 2.233(5) Å. The Cp* moiety is almost perfectly planar as far as the ring atoms are concerned, whereas the methyl groups are bent away from this plane and off from ruthenium.

Experimental

General remarks

All manipulations were performed under an inert atmosphere of argon using *Schlenk* techniques. All chemicals were of standard reagent grade and used without further purification. The solvents were purified according to standard procedures [10]. The deuterated solvents were purchased from Aldrich and dried over 4 Å molecular sieves. [Ru$Cp^*(\mu^3$-Cl)]$_4$, dppm [12], dppe [12], and Ru$Cp^*(dmpe)$Cl [11, 13] were prepared according to the literature. ^{1}H and ^{13}C{^{1}H} NMR spectra were recorded on a Bruker AC-250 spectrometer operating at 250.13 and 62.86 MHz, respectively, and were referenced to internal SiMe$_4$. Microanalyses were performed by the Microanalytical Laboratories, University of Vienna.

{RuCp*(μ-Cl)}₂(μ-dppm) (**1**)

To a solution of [Ru$Cp^*(\mu^3$-Cl)]$_4$ (546 mg, 0.50 mmol) in CH$_2$Cl$_2$, dppm (380 mg, 1.00 mmol, dissolved in CH$_2$Cl$_2$) was slowly added within a period of 2 h. The reaction mixture was stirred for 1 h at room temperature. Insoluble materials were removed by filtration, and the volume of the solution was reduced to about 2 ml. Upon addition of *n*-hexane, an orange-brown precipitate was formed which was collected on a glass-frit and dried under vacuum. **1** was recrystalized from CH$_2$Cl$_2$/Et$_2$O (1:20).

Yield: 714 mg (77%); C$_{45}$H$_{52}$Cl$_2$P$_2$Ru$_2$ (927.91), calc.: C 58.25, H 5.65; found: C 58.38, H 5.89; ^{1}H NMR (δ, CD$_2$Cl$_2$, 20°C): 7.46–7.42 (m, 10H, (*Ph*$_2$P)$_2$CH$_2$), 7.14–7.12 (m, 10H, (*Ph*$_2$P)$_2$CH$_2$), 3.67 (t, $^2J_{HP}$ = 12.7 Hz, 2H, (Ph$_2$P)$_2$C*H*$_2$, 1.25 (s, 30H, *Cp**) ppm; ^{13}C NMR (δ, acetone-d$_6$, 20°C): 133.8 (pt, $^2J_{CP}$ = 5.9 Hz, Ph^{2,6}), 128.8 (Ph^4), 127.5 (pt, $^3J_{CP}$ = 4.4 Hz, Ph^{3,5}), 125.5 (d, $^1J_{CP}$ = 50.2 Hz, Ph^1), 79.5 (*C*$_5$Me$_5$), 29.9 (t, $^1J_{CP}$ = 18.1 Hz, (Ph$_2$P)$_2$C*H*$_2$), 9.35 (C$_5$*Me*$_5$) ppm.

[{RuCp*(μ-Cl)}₂(μ-dppm)](SO₃CF₃)₂ (**2**)

To a solution of **1** (220 mg, 0.237 mmol) in CH$_2$Cl$_2$, AgSO$_3$CF$_3$ (122 mg, 0.475 mmol) was added, whereupon the reaction mixture turned deep red. The mixture was allowed to stir for 1 h at room temperature. Insoluble materials were removed by filtration, and the volume of the solution was reduced to about 1 ml. The product was precipitated by adding Et$_2$O, collected on a glass frit, washed with Et$_2$O, and dried under vacuum.

Yield: 267 mg (92%); C$_{47}$H$_{52}$Cl$_2$F$_6$O$_6$P$_2$Ru$_2$S$_2$ (1226.04), calc.: C 46.05, H 4.28; found: C 45.97, H 4.38; ^{1}H NMR (δ, CD$_3$NO$_2$, 20°C): 7.52–7.25 (m, 20H, (*Ph*$_2$P)$_2$CH$_2$), 3.55 (t, $^2J_{HP}$ = 12.5 Hz, 2H, (Ph$_2$P)$_2$C*H*$_2$, 1.66 ($^4J_{HP}$ = 0.8 Hz, 30H, *Cp**) ppm; ^{13}C NMR (δ, acetone-d$_6$, 20°C): 135.1 (pt, $^2J_{CP}$ = 5.1 Hz, Ph^{2,6}), 133.8 (Ph^4), 130.6 (pt, $^3J_{CP}$ = 4.9 Hz, Ph^{3,5}), 128.9 (d, $^1J_{CP}$ = 53.1 Hz, Ph^1), 112.5 (*C*$_5$Me$_5$), 30.7 (t, $^1J_{CP}$ = 19.1 Hz, (Ph$_2$P)$_2$C*H*$_2$), 11.9 (C$_5$*Me*$_5$) ppm.

{RuCp*(μ-Cl)}₂(μ-dppe) (**3**)

This complex was prepared analogously to **1** with [Ru$Cp^*(\mu^3$-Cl)]$_4$ (546 mg, 0.50 mmol) and dppe (399 mg, 1.00 mmol) as starting materials.

Yield: 565 mg (60%); C$_{46}$H$_{54}$Cl$_2$P$_2$Ru$_2$ (941.64), calc.: C 58.66, H 5.78; found: C 58.74, H 5.89; ^{1}H NMR (δ, CD$_2$Cl$_2$, 20°C): 7.69–7.66 (m, 7H, (*Ph*$_2$P)$_2$CH$_2$CH$_2$), 7.48–7.45 (m, 13H, (*Ph*$_2$P)CH$_2$-CH$_2$), 2.45 (m, 4H, (Ph$_2$P)$_2$C*H*$_2$C*H*$_2$), 2.11 (s, 30H, *Cp**) ppm; ^{13}C NMR (δ aceton-d$_6$, 20°C): 134.8 (pt, $^2J_{CP}$ = 5.6 Hz, Ph^{2,6}), 129.2 (pt, $^3J_{CP}$ = 4.4 Hz, Ph^{3,5}), 128.9 (Ph^4), 126.4 (d, $^1J_{CP}$ = 48.4 Hz, Ph^1), 84.5 (*C*$_5$Me$_5$), 30.2 (m, (Ph$_2$P)$_2$C*H*$_2$C*H*$_2$), 10.2 (C$_5$*Me*$_5$) ppm.

[Ru(Cp*)(dmpe)Cl](SO₃CF₃) (**4**)

A solution of Ru(Cp^*)($dmpe$)Cl (150 mg, 0.356 mmol) in nitromethane was treated with AgCF₃SO₃ (92 mg, 0.356 mmol) at room temperature, whereupon the color of the solution changed immediately from orange to deep red. After removal of the solvent, the residue was dissolved in 2 ml of CH_2Cl_2, and insoluble materials were removed by filtration. Addition of Et_2O afforded a red precipitate which was collected on a glass frit, washed with Et_2O, and dried under vacuum.

Yield: 181 mg (89%); $C_{17}H_{31}ClF_3O_3P_2RuS$ (570.96); calc.: C 35.76, H 5.47; found: C 35.79, H 5.60.

X-Ray structure determination of **2**, **3**, and **4**

Crystal data and experimental details are given in Table 2. X-Ray data were collected on a Philips PW1100 four-circle diffractometer using graphite monochromated MoK_α($\lambda = 0.1069$ Å) radiation and the $\Theta - 2\Theta$ scan technique. Three representative reference reflections were measured every 120 min and used to correct for crystal decay and system instability. Corrections for *Lorentz* and

Table 2. Crystallographic data for $[\{RuCp^*(\mu\text{-Cl})\}_2(\mu\text{-}dppm)](SO_3CF_3)_2$ (**2**), $\{RuCp^*(\mu\text{-Cl})\}_2$-($\mu\text{-}dppe$) (**3**), and $[Ru(Cp^*)(dmpe)Cl](SO_3CF_3)$ (**4**)

	2	**3**	**4**
Formula	$C_{47}H_{52}Cl_2F_6O_6P_2Ru_2S_2$	$C_{46}H_{54}Cl_2P_2Ru_2$	$C_{17}H_{31}ClF_3O_3P_2RuS$
FW	1126.04	941.64	570.94
Cryst. size (mm)	0.05 × 0.27 × 0.29	0.12 × 0.25 × 0.60	0.07 × 0.38 × 0.46
Space group	P2₁/c (No. 15)	P2₁/c (No. 14)	C2 (No. 5)
a (Å)	14.492(3)	18.878(4)	27.780(5)
b (Å)	11.955(3)	10.600(4)	8.041(2)
c (Å)	29.237(5)	23.207(6)	10.781(3)
β (deg)	98.58(1)	108.07(1)	93.11(2)
V (Å³)	5009(2)	4415(2)	2404.7(10)
$F(000)$	2480	1928	1164
Z	4	4	4
ρ_{calc}, (g·cm⁻³)	1.626	1.417	1.577
T(K)	297	297	295
μ (mm⁻¹, Mo K_α)	0.926	0.908	1.021
θ_{max} (deg)	23	23.03	25.03
Index ranges	$-15 \le h \le 15$	$0 \le h \le 20$	$-32 \le h \le 32$
	$0 \le k \le 13$	$0 \le k \le 11$	$0 \le k \le 9$
	$0 \le l \le 32$	$-25 \le l \le 24$	$0 \le l \le 12$
No. of rflns. measd.	7242	6361	2288
No. of unique rflns.	6961	6158	2288
No. of rflns. $F > 4\sigma(F)$	4880	4707	2207
No. of parameters	614	470	263
$R(F)(F > 4\sigma(F))$	0.052	0.041	0.025
$R(F)$ (all data)	0.086	0.064	0.026
$wR(F^2)$ (all data)	0.137	0.084	0.065
Diff. Four peaks min/max (eÅ⁻³)	−0.42/0.60	−0.41/0.37	−0.69/0.69

$R(F) = \sum_o \|F_o| - |F_c\|/\sum |F_o|, wR(F^2) = [\sum(w(F_o^2 - F_c^2)^2)/\sum(w(F_o^2)^2)]^{1/2}$

polarization effects and, where necessary, for absorption were applied. The structures were solved by direct methods [14]. All non-hydrogen atoms were refined anisotropically; hydrogen atoms were included in idealized positions [15]. The structures were refined against F^2. Additional material to the structure determination may be ordered from Fachinformationszentrum Karlsruhe, Gesellschaft für wissenschaftlich-technische Information mbH, D-76344 Eggenstein-Leopoldshafen, Federal Republic of Germany, refering to the deposition number CSD-XXXXX, the names of the authors, and the citation of the present paper.

Acknowledgements

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